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DETAILED DESCRIPTION

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## [Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention relates to the closed mold nonaqueous electrolyte rechargeable battery of high capacity which improved shelf life.

[0002]

[Description of the Prior Art] In recent years, compared with a conventional nickel cadmium cell and a conventional nickel hydride battery, use of the rechargeable lithium-ion battery of high capacity is increasing quickly with the spread of portable electronic equipment, such as a notebook type personal computer and a cellular phone.

[0003] The time of the portable electronic equipment which can be used by one charge is becoming long gradually by high capacity-ization of a cell. On the other hand, since a rechargeable battery deteriorates gradually in order to repeat charge and discharge according to electrochemical reaction, there is a limit in prolonged use. For this reason, there are some which recommend exchanging a cell pack for a new pack in about one year after the beginning of using of a cell pack depending on a pocket device. Since the progress of portable electronic equipment is remarkable, it has been replaced with the new model at the early thing in about three years. As for the cell carried in these devices, it is desirable that it is equivalent to the life of a device. The cause of degradation of a rechargeable battery with the passage of time considers disassembly of the electrolyte in the collapse of structure of the electrode material by the repeat of charge and discharge, and the severe oxidation reduction state in an electrode interface, the time of cell assembly, the influence of moisture which invades from the obturation section after that, etc. While development of an electrode material with sufficient shelf life and selection of a suitable electrolyte are performed, the attempt which lessens the moisture content in a cell is also performed wholeheartedly. Since especially existence of moisture makes the lithium salt in an electrolyte decompose, the moisture management like a cell assembler is important preferably. For example, the electrode sheet of positive/negative is heat-treated before cell assembly, and, usually sufficient dehydration is performed. Moreover, it assembles, after cell assembly material, such as a cell can, is also dehydrated, and it is put on a process. Moreover, it is manufactured using the raw material which also dehydrated the electrolyte, and, usually dehydration is repeated by cell assembly. However, conventionally, even if it such fully dehydrated, degradation with the passage of time may be caused during preservation, and the cause was not solved but it was anxious for solution of this point.

[0004]

[Problem(s) to be Solved by the Invention] The purpose of this invention is excelling in shelf life and offering the nonaqueous electrolyte rechargeable battery which is high capacity.

[0005]

[Means for Solving the Problem] The purpose of this invention was attained by the nonaqueous electrolyte rechargeable battery which made the amount of the free acid contained in this nonaqueous electrolyte after prolonged progress below the specified quantity in the nonaqueous electrolyte rechargeable battery of the closed mold using the nonaqueous electrolyte containing lithium salt.

Namely, this invention is set after pouring in the nonaqueous electrolyte containing (1) lithium salt to the nonaqueous electrolyte rechargeable battery of the closed mold which comes to obturate. The nonaqueous electrolyte rechargeable battery characterized by being the amount in which the amount of the free acid contained in this nonaqueous electrolyte does not exceed 300 ppm after passing through three years after obturation, (2) A nonaqueous electrolyte rechargeable battery given in (1) term characterized by being lithium salt in which the lithium salt contained in nonaqueous electrolyte contains a fluorine, (3) (1) characterized by the free acid contained in this nonaqueous electrolyte being hydrogen fluoride, or a nonaqueous electrolyte rechargeable battery given in (2) terms, (4) (1) to which the gasket used for the obturation section is characterized by carrying out a forming postheat treatment, (2) Or a nonaqueous electrolyte rechargeable battery given in any 1 term of (1) - (4) term characterized by the amount of the free acid which nonaqueous electrolyte before pouring into a nonaqueous electrolyte rechargeable battery given in (3) terms and (5) cell cans contains being 100 ppm or less, (6) A nonaqueous electrolyte rechargeable battery given in any 1 term of (1) - (5) term characterized by the moisture content which the electrode group before inserting in a cell can contains being 300 ppm or less, And any 1 term of (1) - (6) term characterized by for the environment at the time of cell can insertion of (7) this electrode group, electrolytic-solution pouring, and cell can obturation having been managed by less than [ dew-point minus 50 degree C ], and assembling it is provided with the nonaqueous electrolyte rechargeable battery of a publication.

[0006] In this invention, the sum total of the acid inside the cell which a free acid points out the acid detected from the electrolytic solution, and is extracted by the acid contained in the electrolytic solution from the beginning, and the electrolytic solution or its steam, consequently is detected from the electrolytic solution is said. Although the free acid was built in the cell as an acid at the time of cell assembly, it may have generated according to the case and the chemical reaction, for example, it is hydrogen fluoride. Moreover, it says assembling a cell as passing through three years after obturation, and saving for three years under the usual environment (usually temperature of 10-35 degrees C) after obturation. This cell may pass through the process which combined aging processing, charge-and-discharge processing, a charge-and-discharge cycle, and these after obturation if needed.

[0007]

[Embodiments of the Invention] Hereafter, the composition of this invention is explained in full detail. The nonaqueous electrolyte rechargeable battery of this invention obturates the swirl type electrode group which carried out the laminating of the separator and wound it after pouring insertion and an electrolyte into a cell can after electrode sheet manufacture of positive/negative, and comes to carry out aging processing suitable after that. Although the above explained the cylindrical cell, this invention is not restricted to cylindrical but is applied to the cell of arbitrary closed mold, such as a square shape. The nonaqueous electrolyte rechargeable battery of this invention is a cell characterized by being the amount in which the amount of the free acid contained in this nonaqueous electrolyte does not exceed 300 ppm, after passing through three years after obturation. If it is 300 ppm or less, as few, it is desirable, and the amount of a free acid is more desirable if it is 200 ppm or less, and especially if it is 150 ppm or less, it is more desirable. About the operation of the free acid generated [ which is generated and minute-amount-exists ] in a nonaqueous electrolyte rechargeable battery, it was conventionally unknown. The amount of free acids can be determined by carrying out the fixed quantity of the amount of hydrogen fluoride which disassembles a cell and is contained in an electrolyte. The fixed quantity of the amount of hydrogen fluoride uses the bromthymol blue as an indicator, and using decinormal NaOH solution, it can carry out a neutralization titration and it can measure it. Measurement of moisture can be measured with commercial curl Fischer water measurement equipment (for example, Kyoto tradename MKC-210 type curl Fischer water measurement equipment made from an electron).

[0008] In order to suppress the amount of free acids generated in three years after obturation, while making enough dehydration of the component of the cell inserted in a cell can before obturation, it is necessary to raise the degree of sealing of the cell by obturation. After [ fabrication ] annealing (heat treatment) of the gasket for obturation used by this invention is carried out, and it removes the distortion at the time of obturation. As the quality of the material of the gasket which can be used by this

invention, it is olefin system polymer, fluorine system polymer, polyester, cellulose system polymer, a polyimide, and a polyamide, and from organic-solvent-proof nature and low-water-flow part permeability, olefin system polymer and polyester are desirable and the block copolymerization polymer of polypropylene, a propylene, and ethylene and a polybutylene terephthalate are especially desirable. The gasket used for the nonaqueous electrolyte rechargeable battery of this invention is heat-treated after that, although fabricated by the configuration which can carry out caulking obturation with obturation members, such as a cover-distributor end and a relief valve. If heat treatment is not enough, it will become the cause by which deformation takes place gradually and the degree of sealing falls after caulking \*\*. The temperature of heat treatment has desirable 60-130 degrees C, its 80-120 degrees C are more desirable, and especially its 90-110 degrees C are desirable. The time of heat treatment has 10 minutes - 5 desirable hours, and 20 minutes - especially its 2 hours are desirable.

[0009] As for the nonaqueous electrolyte used for the nonaqueous electrolyte rechargeable battery of this invention, it is desirable that the amount of the free acid contained before pouring into a cell can is 100 ppm or less. The amount of a more desirable free acid is 60 ppm or less, and especially a desirable thing is 40 ppm or less. In order to make the electrolytic solution with few moisture and the amounts of free acids, it is required to use what has the high purity of a supporting electrolyte with repeating distillation, or making dehydrating agents, such as a molecular sieve, act, and fully dehydrating an electrolysis solvent. It is desirable that a dew-point performs manufacture of the electrolytic solution in the dry air not more than minus 50 degree C or inert gas. You may dehydrate the created electrolytic solution further.

[0010] Generally the electrolytic solution consists of a supporting electrolyte and a solvent. As for the supporting electrolyte in a lithium secondary battery, lithium salt is mainly used. As lithium salt which can be used for the nonaqueous electrolyte rechargeable battery of this invention For example,  $\text{LiClO}_4$ ,  $\text{LiBF}_4$ ,  $\text{LiPF}_6$ , and  $\text{LiCF}_3\text{CO}_2$ ,  $\text{LiAsF}_6$ ,  $\text{LiSbF}_6$ ,  $\text{LiB}_{10}\text{Cl}_{10}$ , the fluorosulfonic acid expressed with  $\text{LiOSO}_2\text{C}_n\text{F}_{2n+1}$  ( $n$  is six or less positive integer), The imido salt expressed with  $\text{LiN}(\text{SO}_2\text{C}_n\text{F}_{2n+1})(\text{SO}_2\text{C}_m\text{F}_{2m+1})$  ( $m$  and  $n$  are six or less positive integer, respectively), The methide salt expressed with  $\text{LiN}(\text{SO}_2\text{C}_p\text{F}_{2p+1})(\text{SO}_2\text{C}_q\text{F}_{2q+1})(\text{SO}_2\text{C}_r\text{F}_{2r+1})$  ( $p$ ,  $q$ , and  $r$  are six or less positive integer, respectively), Li salts, such as a low-grade aliphatic carboxylic-acid lithium,  $\text{LiAlCl}_4$ ,  $\text{LiCl}$ ,  $\text{LiBr}$ ,  $\text{LiI}$ , a chloro borane lithium, and 4 phenyl lithium borate, can be mentioned, and these kinds or two sorts or more can be mixed and used. It is  $\text{LiBF}_4$  especially. And/or  $\text{LiPF}_6$  What was dissolved is desirable. Although especially the concentration of a supporting electrolyte is not limited, its 0.2-3 mols per 1l. of electrolytic solutions are desirable.

[0011] As a solvent which can be used for the nonaqueous electrolyte rechargeable battery of this invention Propylene carbonate, ethylene carbonate, butylene carbonate, Dimethyl carbonate, diethyl carbonate, methylethyl carbonate, Gamma-butyrolactone, methyl formate, methyl acetate, 1, 2-dimethoxyethane, A tetrahydrofuran, 2-methyl tetrahydrofuran, dimethyl sulfoxide, 1, 3-dioxolane, a formamide, a dimethylformamide, a dioxolane, A dioxane, an acetonitrile, a nitromethane, an ethyl monochrome glyme, Trialkyl phosphate, trimethoxy methane, a dioxolane derivative, a sulfolane, Non-proton nature organic solvents, such as 3-methyl-2-oxazolidinone, propylene carbonate derivative, tetrahydrofuran derivative, ethyl ether, 1, and 3-propane ape ton, can be mentioned, and these kinds or two sorts or more are mixed and used. In these, the solvent of a carbonate system is desirable and it is desirable especially to mix and use annular carbonate and un-annular carbonate. As annular carbonate, ethylene carbonate and propylene carbonate are desirable. Moreover, as un-annular carbonate, diethyl carbonate, dimethyl carbonate, and methylethyl carbonate are desirable.

[0012] As the electrolytic solution which can be used for the nonaqueous electrolyte rechargeable battery of this invention, they are  $\text{LiCF}_3\text{SO}_3$ ,  $\text{LiClO}_4$ , and  $\text{LiBF}_4$  to the electrolytic solution which mixed suitably ethylene carbonate, propylene carbonate, 1, 2-dimethoxyethane, dimethyl carbonate, or diethyl carbonate. And/or,  $\text{LiPF}_6$  The included electrolytic solution is desirable. To one [ at least ] mixed solvent of propylene carbonate, at least one side of ethylene carbonate and dimethyl carbonate, or diethyl carbonate, they are  $\text{LiCF}_3\text{SO}_3$ ,  $\text{LiClO}_4$ , or  $\text{LiBF}_4$  especially. At least a kind of salt and  $\text{LiPF}_6$  which were chosen from inside The included electrolytic solution is desirable. Especially the amount

that adds these electrolytic solutions in a cell is not limited, but can be used according to the amount of positive-electrode material or negative-electrode material, or the size of a cell.

[0013] Moreover, the following solid electrolytes can also be used together besides the electrolytic solution. It is divided into an inorganic solid electrolyte and an organic solid electrolyte as a solid electrolyte. The nitride of Li, the halogenide, the oxygen-acid salt, etc. are well known by the inorganic solid electrolyte. Especially,  $\text{Li}_3\text{N}$ ,  $\text{LiI}$ ,  $\text{Li}_5\text{NI}_2$ ,  $\text{Li}_3\text{N-LiI-LiOH}$ ,  $\text{Li}_4\text{SiO}_4$ ,  $\text{Li}_4\text{SiO}_4\text{-LiI-LiOH}$ ,  $x\text{Li}_3\text{PO}_4\text{-(1-x)Li}_4\text{SiO}_4$ ,  $\text{Li}_2\text{SiS}_3$ , a phosphorus-sulfide compound, etc. are effective (being here  $0 < x \leq 1$ ).

[0014] In an organic solid electrolyte, the macromolecule matrix material which made the polymer containing the polymer, the polypropylene oxide derivative, or this derivative containing a polyethylene-oxide derivative or this derivative, the polymer containing an ionic dissociation machine, the polymer containing an ionic dissociation machine, the mixture of the above-mentioned non-proton nature electrolytic solution and phosphoric ester polymer, and the non-proton nature polar solvent contain is effective. Furthermore, there is also the method of adding a polyacrylonitrile to the electrolytic solution. Moreover, how to use inorganic and an organic solid electrolyte together is also learned.

[0015] Moreover, you may add other compounds to an electrolyte for the purpose which improves electric discharge and a charge-and-discharge property. For example, a pyridine, pyrroline, a pyrrole, a triphenylamine, a phenyl carbazole, A triethyl force fight, a triethanolamine, cyclic ether, Ethylenediamine, hexalin acid TORIAMIDO, a nitrobenzene derivative, Sulfur, a quinonimine dye, N-substitution oxazolidinone, and N and N'-substitution imidalidinone, Glymes, for example, ethylene glycol dialkyl ether, quaternary ammonium salt, A polyethylene glycol, a pyrrole, a 2-methoxyethanol, and  $\text{AlCl}_3$ , The monomer of a conductive polymer electrode active material, the triethylenephosphoramidate, An aryl compound with trialkylphosphine, a morpholine, and a carbonyl group, The crown ethers like 12-crown -4, hexamethylphosphoric triamide and 4-alkyl morpholine, the bicyclic third class amine, oil, the fourth class phosphonium salt, the third class sulfonium salt, etc. can be mentioned. Especially a desirable thing is independent or the case where it combined and uses, about a triphenylamine and a phenyl carbazole.

[0016] Moreover, in order to make the electrolytic solution into incombustibility, a \*\* halogen solvent, for example, a carbon tetrachloride, and 3 fluoride-salt-ized ethylene can be included in the electrolytic solution. Moreover, in order to give fitness to elevated-temperature preservation, carbon dioxide gas can be included in the electrolytic solution.

[0017] Although the electrolytic solution may pour in the whole quantity at once, it is desirable to pour in in 2 steps or more. When pouring in in 2 steps or more, the composition (after pouring in the solution which dissolved lithium salt in the non-aqueous solvent or the non-aqueous solvent, the solution which dissolved lithium salt in a non-aqueous solvent or a non-aqueous solvent with viscosity higher than the aforementioned solvent is poured in) which is different also by the same composition is sufficient as each liquid. Moreover, a cell can may be decompressed for shortening of the pouring time of the electrolytic solution etc., or you may perform applying a centrifugal force and an ultrasonic wave to a cell can.

[0018] As for the electrode group of the nonaqueous electrolyte rechargeable battery of this invention, it is desirable that the moisture content which the electrode group before inserting in a cell can contains is 300 ppm or less. 200 ppm or less are more desirable, and especially 100 ppm or less are desirable. obturation of the material which constitutes cells other than an electrode group, for example, a cell can, an electric insulating plate, a gasket, etc., etc. -- it is desirable that a member etc. fully dehydrates before cell assembly After washing these members using a suitable solvent etc., it is desirable that a dew-point keeps it in the dry air not more than minus 50 degree C or inert gas.

[0019] The electrode sheet after an application is dried and dehydrated by hot blast, a vacuum, infrared radiation, far infrared rays, an electron ray, and the operation of the damp style. these methods are independent -- or it can combine and use The range of drying temperature of 50-350 degrees C is desirable. After heat-treating at 150-260 degrees C after more specifically carrying out press forming of the electrode sheet dried at 50-100 degrees C, and judging to the operating width of face of an electrode, it is desirable to dehydrate further at 160-250 degrees C before cell assembly. As for heat treatment, it is

desirable especially to process at the temperature of 190-250 degrees C for 2 minutes to 2 hours, and, as for dehydration before cell assembly, it is desirable especially to process at the temperature of 170-240 degrees C for 20 minutes to 10 hours. The moisture content which the electrode in front of cell assembly has has desirable 200 ppm or less, and especially its 100 ppm or less are desirable.

[0020] As for the assembly environment of a cell, in manufacture of the nonaqueous electrolyte rechargeable battery of this invention, it is desirable to be managed by less than [ dew-point minus 50 degree C ] through each process of cell can insertion of this electrode group, electrolytic-solution pouring, and cell can obturation.

[0021] the positive electrode (or negative electrode) used for the nonaqueous electrolyte rechargeable battery of this invention -- a positive electrode -- on a charge collector, a mixture (or a negative electrode mixture) can be painted and fabricated, and can be made a positive electrode -- an others and electric conduction agent, a binder, a dispersant, a filler, an ion electric conduction agent, a pressure reinforcement agent, and various additives can be included in a mixture (or a negative electrode mixture) [ positive active material / (or negative-electrode material) ] Although these electrodes may be the shape of a disk, and a tabular, they are desirable in having the shape of a supple sheet.

[0022] the following -- the electrode of the nonaqueous electrolyte rechargeable battery of this invention -- the material used for a mixture is explained The positive active material used for the nonaqueous electrolyte rechargeable battery of this invention has a desirable lithium content transition-metals oxide. A lithium content transition-metals oxide is an oxide which mainly contains at least one sort of transition-metals elements chosen from Ti, V, Cr, Mn, Fe, Co, nickel, Mo, and W, and a lithium, and the mole ratio of a lithium and transition metals is the compound of 0.3 or 2.2. It is the oxide which mainly contains more preferably at least one sort of transition-metals elements chosen from V, Cr, Mn, Fe, Co, and nickel, and a lithium, and the mole ratio of a lithium and transition metals is the compound of 0.3 or 2.2. In addition, you may contain aluminum, Ga, In, germanium, Sn, Pb, Sb, Bi, Si, P, B, etc. in less than 30 mole percents to the transition metals which mainly exist. A still more desirable lithium content transition-metals oxide  $\text{Li}_x\text{CoO}_2$ ,  $\text{Li}_x\text{NiO}_2$ ,  $\text{Li}_x\text{MnO}_2$ , and  $\text{Li}_x\text{Coa nickel1-a O}_2$ ,  $\text{Li}_x\text{Cob V1-b O}_2$  and  $\text{Li}_x\text{Cob Fe1-b O}_2$ ,  $\text{Li}_x\text{Mn 2O}_4$ ,  $\text{Li}_x\text{Mnc Co2-c O}_4$ , and  $\text{Li}_x\text{Mnc nickel2-c O}_4$ , They are  $\text{Li}_x\text{Mnc V2-c O}_4$  and  $\text{Li}_x\text{Mnc Fe2-c O}_4$  (it is  $x=0.02-1.2$ ,  $a=0.1$  to  $0.9$ ,  $b=0.8$  to  $0.98$ ,  $c=1.6-1.96$ , and  $z=2.01-2.3$  here). As most desirable lithium content transition-metals oxide,  $\text{Li}_x\text{CoO}_2$ ,  $\text{Li}_x\text{NiO}_2$ ,  $\text{Li}_x\text{MnO}_2$ ,  $\text{Li}_x\text{Coa nickel1-a O}_2$ ,  $\text{Li}_x\text{Mn 2O}_4$ , and  $\text{Li}_x\text{Cob V1-b O}_2$  ( $x=0.02-1.2$ ,  $a=0.1$  to  $0.9$ ,  $b=0.9$  to  $0.98$ ,  $z=2.01-2.3$ ) are raised. In addition, the value of  $x$  is a value before a charge-and-discharge start, and is fluctuated by charge and discharge.

[0023] Although the positive active material used for the nonaqueous electrolyte rechargeable battery of this invention is compoundable with the method and solution reaction which mix a lithium compound and a transition-metals compound and are calcinated, especially its calcinating method is desirable. The detail for baking is indicated by the paragraph 35 of JP,6-60867,A, JP,7-14579,A, etc., and these methods can be used for it. After washing by water, acid solution, alkaline solution, and the organic solvent, you may use the positive active material obtained by baking. Furthermore, you may be the method of compounding by making it react to a transition-metals oxide with a lithium metal, a lithium alloy, or a butyl lithium and a transition-metals oxide as a method of inserting a lithium ion chemically.

[0024] Although especially the average grain size of the positive active material used for the nonaqueous electrolyte rechargeable battery of this invention is not limited, 0.1-50 micrometers is desirable. It is desirable that the volume of a 0.5-30-micrometer particle is 95% or more. It is still more desirable that the volume which a particle group with a particle size of 3 micrometers or less occupies is 18% or less of a whole product, and the volume which 15-micrometer or more particle group 25 micrometers or less occupies is 18% or less of a whole product. Although not limited especially as a specific surface area,  $0.01-50\text{m}^2/\text{g}$  is especially desirable at a BET adsorption method, and  $0.2\text{m}^2/\text{g}$  is desirable. Moreover, as pH of the supernatant when melting 5g of positive active materials to 100ml of distilled water, 12 or less [ 7 or more ] are desirable.

[0025] When obtaining the positive active material of the nonaqueous electrolyte rechargeable battery of this invention by baking, it is 700-1200 degrees C that it is 500-1500 degrees C as a burning temperature

desirable still more preferably, and it is 750-1000 degrees C especially preferably. As a firing time, 4 - 30 hours is 6 - 20 hours desirable still more preferably, and it is 6 - 15 hours especially preferably.

[0026] As a negative-electrode material used with the nonaqueous electrolyte rechargeable battery of this invention, what is necessary is just occlusion and the compound which can be emitted about a lithium ion. As an example of such a negative-electrode material, a metal lithium, a lithium alloy, a carbonaceous compound, an inorganic oxide, an inorganic chalcogen compound, a metal complex, and an organic high molecular compound are mentioned. Even if these are independent, it may combine and they may use.

[0027] The above-mentioned desirable negative-electrode material is the oxide of carbonaceous material, a metal, or a half-\*\*\*\* element, and chalcogen. Carbonaceous material is a material which consists of carbon substantially, for example, can mention a petroleum pitch, a natural graphite, an artificial graphite, difficulty graphite nature carbon, a meso carbon micro bead, a PAN system carbon fiber, a cellulose system carbon fiber, a pitch based carbon fiber, a vapor-growth carbon fiber, a dehydration PVA system carbon fiber, a lignin carbon fiber, a vitrified carbon fiber, an activated carbon fiber, etc.

[0028] The oxide of the metal used with the nonaqueous electrolyte rechargeable battery of this invention or a half-\*\*\*\* element and a chalcogen compound are compounds which consist of periodic tables 13 and 14, 15 group atom, oxygen, or a chalcogen group atom. It mainly concerns including three or more sorts of atoms chosen from periodic tables 1, 2, 13, and 14 and 15 group atom as a negative-electrode material in this invention, and an amorphous chalcogen compound or an amorphous oxide is used especially preferably. It is the object which has the broadcloth dispersion band which mainly has the peak from 20 degrees to 40 degrees with 2theta value with the X-ray diffraction method [ be / amorphous ] using CuK alpha rays said here, and you may have a crystalline diffraction line. Among the crystalline diffraction lines preferably looked at by 40 degrees or more 70 degrees or less with 2theta value, it is 100 or less times that they are 500 or less times of the diffraction line intensity of the peak of the broadcloth dispersion band looked at by 20 degrees or more 40 degrees or less with 2theta value desirable still more preferably, and the strongest intensity is 5 or less times especially preferably, and is not having the most desirable crystalline diffraction line.

[0029] The above-mentioned chalcogen compound and an oxide have more desirable compound chalcogen compound and multiple oxide which make a subject two or more sorts of elements in B, aluminum, Ga, In, Tl, Si, germanium, Sn, Pb, P, As, Sb, and Bi. Especially a desirable thing is the compound chalcogen compound or oxide which makes a subject two or more sorts of elements in B, aluminum, Si, germanium, Sn, and P. Since amorphous structure is mainly embellished, these compound chalcogen compounds and a multiple oxide contain at least one sort of elements chosen from the element of one to two groups of a periodic table.

[0030] The amorphous multiple oxide which makes Sn a subject in the above-mentioned negative-electrode material is desirable, and is expressed with the following general formula (3).

General formula (3) The inside of a  $\text{SnM}_3\text{cM}_4\text{dOt}$  formula, and M3 It is at least one sort of aluminum, B, P, Si, and germanium M4 Expressing at least one sort of the 1st group element of a periodic table, and the 2nd group element, from 0.2, c is size and less than two number, d is 0.01 or more and 1 or less number, and  $0.2 < c + d < 3$  and t express or more 1 six or less number.

[0031] Although any method of the calcinating method and a solution method can be used for the amorphous multiple oxide used with the nonaqueous electrolyte rechargeable battery of this invention, its calcinating method is more desirable. After often mixing the oxide or compound of an element indicated by the general formula (1) by the calcinating method, it is desirable to calcinate and to obtain an amorphous multiple oxide.

[0032] As baking conditions, it is desirable that it is 200 degrees C or less of 5-degree-C [ or more ]/m programming rates as a programming rate, it is desirable as a burning temperature that it is [ 500 degrees-C or more ] 1500 degrees C or less, and it is desirable that it is 100 or less hours as a firing time for 1 hour or more. And it is desirable that it is below 2-degree-C[ or more ]/m 107 \*\* as a lower temperature fall speed. The programming rate in manufacture of the nonaqueous electrolyte



rechargeable battery of this invention is the mean velocity of a temperature rise until it reaches "80% of burning temperature (degree-C display)" from "50% of burning temperature (degree-C display)", and the temperature fall speed in this invention is the mean velocity of a temperature reduction until it reaches "50% of burning temperature (degree-C display)" from "80% of burning temperature (degree-C display)."

[0033] You may cool in a firing furnace and a temperature fall is taken out out of a firing furnace again, for example, may be supplied underwater and may be cooled. moreover, the gun method, the Hammer-Anvil method, and the slap method given in 217 pages (Gihodo Shuppan Co., Ltd. 1987) of ceramic processing -- the - gas atomizing method, the plasma-spraying method, a centrifugal quenching method, and melt Super-quenching methods, such as the drag method, can also be used. Moreover, you may cool using the single roller method given in 172 pages (Maruzen 1991) of new glass handbooks, and a congruence roller. In the case of the material fused during baking, you may take out a baking object continuously, supplying a raw material during baking. It is desirable to stir a melt in the case of the material fused during baking.

[0034] Oxygen content is the atmosphere below 5 volume % preferably, and baking gas atmosphere is inert gas atmosphere still more preferably. Nitrogen, an argon, helium, a krypton, a xenon, etc. are mentioned as inert gas. The most desirable inert gas is a pure argon.

[0035] The average grain size of the compound used for manufacture of the nonaqueous electrolyte rechargeable battery of this invention has desirable 0.1-60 micrometers. It is desirable that a mean particle diameter is 0.7-25 micrometers, and 60% or more of a whole product is 0.5-30 micrometers in more detail. Moreover, as for the volume which a particle group with a particle size [ of the negative-electrode active material of the nonaqueous electrolyte rechargeable battery of this invention ] of 1 micrometer or less occupies, it is desirable that it is 30% or less of a whole product, and the volume which a particle group with a particle size of 20 micrometers or more occupies is 25% or less of a whole product. the particle size of the material to be used -- the mixture of one side of a negative electrode -- it cannot be overemphasized that it is what does not exceed thickness

[0036] In order to make it a predetermined grain size, the grinder and classifier which were known well are used. For example, a mortar, a ball mill, a sand mill, a vibration ball mill, a satellite ball mill, a planet ball mill, a revolution air current type jet mill, a screen, etc. are used. At the time of pulverization, wet grinding which made organic solvents, such as water or a methanol, live together can also be performed if needed. It is desirable to perform a classification in order to consider as a desired particle size. As the classification method, there is especially no limitation and it can use a screen, a pneumatic elutriation machine, etc. if needed. A classification can use dry type and wet. A mean particle diameter is a median size of a primary particle, and it is measured by the particle-size-distribution measuring device of a laser diffraction formula. Moreover, as for the specific surface area of the negative-electrode material of the nonaqueous electrolyte rechargeable battery of this invention, it is desirable that the measured value in a BET specific surface area measuring method is 0.1-5m<sup>2</sup> / g.

[0037] Although the example of the negative-electrode material of the nonaqueous electrolyte rechargeable battery of this invention is shown below, this invention is not limited to these. SnAl 0.4B0.5P0.5K0.1O3.65 and SnAl0.4 B0.5 P0.5 Na 0.2O3.7, SnAl0.4 B0.3 P0.5 Rb 0.2O3.4, SnAl0.4 B0.5 P0.5 Cs 0.1O3.65, SnAl0.4 B0.5 P0.5 K0.1 germanium 0.05O3.85, and SnAl0.4 B0.5 P0.5 K0.1 Mg0.1 germanium 0.02O3.83, SnAl 0.4B0.4P0.4O3.2 and SnAl 0.3B0.5P0.2O2.7, SnAl 0.3B0.5P0.2O2.7 and SnAl0.4 B0.5 P0.3 Ba0.08Mg 0.08O3.26, SnAl0.4B0.4 P0.4 Ba 0.08O3.28, and SnAl 0.4B0.5P0.5O3.6, SnAl0.4 B0.5 P0.5 Mg 0.1O3.7, [0038] SnAl0.5 B0.4 P0.5 Mg0.1 F0.2 O3.65 and SnB0.5 P0.5 Li0.1 Mg 0.1F0.2O3.05, SnB0.5 P0.5 K0.1 Mg 0.1F0.2O3.05, SnB0.5 P0.5 K0.05Mg0.05F0.1 O3.03, and SnB0.5 P0.5 K0.05Mg0.1 F0.2O3.03 and SnAl0.4 B0.5 P0.5 Cs0.1 Mg0.1 F0.2 O3.65, SnB0.5P0.5 Cs0.05Mg0.05F0.1 O3.03, SnB0.5 P0.5 Mg 0.1F0.1O3.05, and SnB0.5 P0.5 Mg 0.1F0.2O3, SnB0.5 P0.5 Mg 0.1F0.06O3.07, SnB0.5 P0.5 Mg 0.1F0.14O3.03, SnPBa 0.08O3.58, SnPK 0.1O3.55, SnPK0.05Mg0.05O3.58, and SnPCs0.1 O3.55, SnPBa0.08F0.08O3.54, SnPK0.1 Mg 0.1F0.2O3.55, SnPK0.05Mg0.05F0.1 O3.53, SnPCs0.1 Mg 0.1F0.2O3.55, SnPCs0.05Mg0.05F0.1 O3.53, [0039] Sn1.1 aluminum 0.4B0.2P0.6 Ba0.08F0.08O3.54 and Sn1.1

aluminum<sub>0.4</sub> B<sub>0.2</sub> P<sub>0.6</sub> Li<sub>0.1</sub> K<sub>0.1</sub> Ba<sub>0.1</sub> F<sub>0.1</sub> O<sub>3.65</sub> and Sn<sub>1.1</sub> aluminum<sub>0.4</sub> B<sub>0.4</sub> P<sub>0.4</sub> Ba<sub>0.08</sub> O<sub>3.34</sub>, Sn<sub>1.1</sub> aluminum<sub>0.4</sub> P<sub>0.05</sub> O<sub>4.23</sub>, Sn<sub>1.1</sub> aluminum<sub>0.4</sub> PK<sub>0.05</sub> O<sub>4.23</sub>, and Sn<sub>1.2</sub> aluminum<sub>0.5</sub> B<sub>0.3</sub> P<sub>0.4</sub> Cs<sub>0.2</sub> O<sub>3.5</sub>, Sn<sub>1.2</sub> aluminum<sub>0.4</sub> B<sub>0.2</sub> P<sub>0.6</sub> Ba<sub>0.08</sub> F<sub>0.08</sub> O<sub>3.64</sub>, Sn<sub>1.2</sub> aluminum<sub>0.4</sub> B<sub>0.2</sub> P<sub>0.6</sub> Mg<sub>0.04</sub> Ba<sub>0.04</sub> O<sub>3.68</sub>, Sn<sub>1.2</sub> aluminum<sub>0.4</sub> B<sub>0.3</sub> P<sub>0.5</sub> Ba<sub>0.08</sub> O<sub>3.58</sub>, and Sn<sub>1.3</sub> aluminum<sub>0.3</sub> B<sub>0.3</sub> P<sub>0.4</sub> Na<sub>0.2</sub> O<sub>3.3</sub> and Sn<sub>1.3</sub> aluminum<sub>0.2</sub> B<sub>0.4</sub> P<sub>0.4</sub> calcium<sub>0.2</sub> O<sub>3.4</sub>, Sn<sub>1.3</sub> aluminum<sub>0.4</sub> B<sub>0.4</sub> P<sub>0.4</sub> Ba<sub>0.2</sub> O<sub>3.6</sub>, Sn<sub>1.4</sub> aluminum<sub>0.4</sub> PK<sub>0.2</sub> O<sub>4.6</sub>, Sn<sub>1.4</sub> aluminum<sub>0.2</sub> Ba<sub>0.1</sub> PK<sub>0.2</sub> O<sub>4.45</sub>, and Sn<sub>1.4</sub> aluminum<sub>0.2</sub> Ba<sub>0.2</sub> PK<sub>0.2</sub> O<sub>4.6</sub>, Sn<sub>1.4</sub> aluminum<sub>0.4</sub> Ba<sub>0.2</sub> PK<sub>0.2</sub> Ba<sub>0.1</sub> F<sub>0.2</sub> O<sub>4.9</sub>, Sn<sub>1.4</sub> aluminum<sub>0.4</sub> PK<sub>0.3</sub> O<sub>4.65</sub> and Sn<sub>1.5</sub> aluminum<sub>0.2</sub> PK<sub>0.2</sub> O<sub>4.4</sub>, Sn<sub>1.5</sub> aluminum<sub>0.4</sub> PK<sub>0.1</sub> O<sub>4.65</sub>, Sn<sub>1.5</sub> aluminum<sub>0.4</sub> P<sub>0.05</sub> O<sub>4.63</sub>, Sn<sub>1.5</sub> aluminum<sub>0.4</sub> P<sub>0.05</sub> Mg<sub>0.1</sub> F<sub>0.2</sub> O<sub>4.63</sub>, [0040] SnSi<sub>0.5</sub> aluminum<sub>0.1</sub> B<sub>0.2</sub> P<sub>0.1</sub> calcium<sub>0.4</sub> O<sub>3.1</sub>, SnSi<sub>0.4</sub> aluminum<sub>0.2</sub> B<sub>0.4</sub> O<sub>2.7</sub> and SnSi<sub>0.5</sub> aluminum<sub>0.2</sub> B<sub>0.1</sub> P<sub>0.1</sub> Mg<sub>0.1</sub> O<sub>2.8</sub>, SnSi<sub>0.6</sub> aluminum<sub>0.2</sub> B<sub>0.2</sub> O<sub>2.8</sub>, SnSi<sub>0.5</sub> aluminum<sub>0.3</sub> B<sub>0.4</sub> P<sub>0.2</sub> O<sub>3.55</sub> and SnSi<sub>0.5</sub> aluminum<sub>0.3</sub> B<sub>0.4</sub> P<sub>0.5</sub> O<sub>4.30</sub> and SnSi<sub>0.6</sub> aluminum<sub>0.1</sub> B<sub>0.1</sub> P<sub>0.3</sub> O<sub>3.25</sub>, SnSi<sub>0.6</sub> aluminum<sub>0.1</sub> B<sub>0.1</sub> P<sub>0.1</sub> Ba<sub>0.2</sub> O<sub>2.95</sub>, SnSi<sub>0.6</sub> aluminum<sub>0.1</sub> B<sub>0.1</sub> P<sub>0.1</sub> calcium<sub>0.2</sub> O<sub>2.95</sub>, and SnSi<sub>0.6</sub> aluminum<sub>0.4</sub> B<sub>0.2</sub> Mg<sub>0.1</sub> O<sub>3.2</sub>, SnSi<sub>0.6</sub> aluminum<sub>0.1</sub> B<sub>0.3</sub> P<sub>0.1</sub> O<sub>3.05</sub>, and SnSi<sub>0.6</sub> aluminum<sub>0.2</sub> Mg<sub>0.2</sub> O<sub>2.7</sub>, SnSi<sub>0.6</sub> aluminum<sub>0.2</sub> calcium<sub>0.2</sub> O<sub>2.7</sub>, SnSi<sub>0.6</sub> aluminum<sub>0.2</sub> P<sub>0.2</sub> O<sub>3</sub>, and SnSi<sub>0.6</sub> B<sub>0.2</sub> P<sub>0.2</sub> O<sub>3</sub>, SnSi<sub>0.8</sub> aluminum<sub>0.2</sub> O<sub>2.9</sub>, SnSi<sub>0.8</sub> aluminum<sub>0.3</sub> B<sub>0.2</sub> P<sub>0.2</sub> O<sub>3.85</sub>, and SnSi<sub>0.8</sub> B<sub>0.2</sub> O<sub>2.9</sub>, SnSi<sub>0.8</sub> Ba<sub>0.2</sub> O<sub>2.8</sub>, SnSi<sub>0.8</sub> Mg<sub>0.2</sub> O<sub>2.8</sub>, SnSi<sub>0.8</sub> calcium<sub>0.2</sub> O<sub>2.8</sub>, SnSi<sub>0.8</sub> P<sub>0.2</sub> O<sub>3.1</sub>, [0041] Sn<sub>0.9</sub> Mn<sub>0.3</sub> B<sub>0.4</sub> P<sub>0.4</sub> calcium<sub>0.1</sub> Rb<sub>0.1</sub> O<sub>2.95</sub> and Sn<sub>0.9</sub> Fe<sub>0.3</sub> B<sub>0.4</sub> P<sub>0.4</sub> calcium<sub>0.1</sub> Rb<sub>0.1</sub> O<sub>2.95</sub> and Sn<sub>0.8</sub> Pb<sub>0.2</sub> calcium<sub>0.1</sub> P<sub>0.9</sub> O<sub>3.35</sub> and Sn<sub>0.3</sub> germanium<sub>0.7</sub> Ba<sub>0.1</sub> P<sub>0.9</sub> O<sub>3.35</sub> and Sn<sub>0.9</sub> Mn<sub>0.1</sub> Mg<sub>0.1</sub> P<sub>0.9</sub> O<sub>3.35</sub>, Sn<sub>0.2</sub> Mn<sub>0.8</sub> Mg<sub>0.1</sub> P<sub>0.9</sub> O<sub>3.35</sub>, Sn<sub>0.7</sub> Pb<sub>0.3</sub> calcium<sub>0.1</sub> P<sub>0.9</sub> O<sub>3.35</sub>, Sn<sub>0.2</sub> germanium<sub>0.8</sub> Ba<sub>0.1</sub> P<sub>0.9</sub> O<sub>3.35</sub> [0042] SnSi<sub>0.8</sub> B<sub>0.2</sub> O<sub>2.9</sub>, SnSi<sub>0.7</sub> B<sub>0.3</sub> O<sub>2.85</sub>, and SnSi<sub>0.7</sub> B<sub>0.3</sub> aluminum<sub>0.1</sub> O<sub>3.0</sub>, SnSi<sub>0.5</sub> B<sub>0.3</sub> aluminum<sub>0.1</sub> Mg<sub>0.1</sub> O<sub>2.7</sub>, Sn<sub>0.8</sub> Si<sub>0.6</sub> B<sub>0.2</sub> aluminum<sub>0.1</sub> Li<sub>0.1</sub> O<sub>2.5</sub>, Sn<sub>0.8</sub> Si<sub>0.6</sub> B<sub>0.2</sub> aluminum<sub>0.1</sub> Cs<sub>0.1</sub> O<sub>2.65</sub>, Sn<sub>0.8</sub> Si<sub>0.7</sub> B<sub>0.1</sub> P<sub>0.1</sub> aluminum<sub>0.1</sub> O<sub>2.75</sub>, and Sn<sub>0.8</sub> Si<sub>0.5</sub> B<sub>0.3</sub> P<sub>0.2</sub> aluminum<sub>0.1</sub> O<sub>2.9</sub>, Sn<sub>0.8</sub> Si<sub>0.7</sub> B<sub>0.1</sub> P<sub>0.1</sub> aluminum<sub>0.1</sub> Li<sub>0.05</sub> O<sub>2.78</sub> and Sn<sub>0.8</sub> Si<sub>0.5</sub> B<sub>0.3</sub> P<sub>0.1</sub> aluminum<sub>0.1</sub> Li<sub>0.1</sub> O<sub>2.7</sub>, Sn<sub>0.8</sub> Si<sub>0.5</sub> B<sub>0.3</sub> P<sub>0.2</sub> aluminum<sub>0.1</sub> Cs<sub>0.1</sub> O<sub>2.95</sub>, Sn<sub>0.8</sub> Si<sub>0.7</sub> P<sub>0.3</sub> O<sub>2.95</sub>, and Sn<sub>0.8</sub> Si<sub>0.7</sub> P<sub>0.3</sub> aluminum<sub>0.1</sub> O<sub>3.1</sub>, SnSi<sub>0.5</sub> B<sub>0.3</sub> Zr<sub>0.1</sub> O<sub>2.65</sub>, Sn<sub>0.8</sub> Si<sub>0.6</sub> P<sub>0.2</sub> Zr<sub>0.1</sub> O<sub>2.7</sub>, Sn<sub>0.8</sub> Si<sub>0.6</sub> B<sub>0.2</sub> P<sub>0.1</sub> Zr<sub>0.1</sub> O<sub>2.75</sub> [0043] The chemical formula of the compound which baking was carried out [ above-mentioned ] and obtained is computable from the weight difference of the fine particles before and behind baking as inductively-coupled-plasma (ICP) emission spectrochemical analysis and a shortcut method as a measuring method.

[0044] A light metal, especially a lithium can be inserted and used for the negative-electrode material of the nonaqueous electrolyte rechargeable battery of this invention. There are electrochemical process, the chemical method, the thermal method, etc. as insertion method of a lithium. especially a desirable thing - - electrochemical process -- it is -- for example, the negative electrode of a charge collector -- the wafer of the metal which made the lithium the subject is stuck on the uncoated portion and negative-electrode binder layer of a mixture, and it can insert by making the electrolytic solution contact The method of inserting a lithium electrochemically especially within a cell is desirable. As for the wafer of the metal which made the lithium the subject, it is desirable to stick by making the foil whose thickness is 5-200 micrometers into wafers, such as the shape of a strip of paper.

[0045] Insertion of a lithium can be inserted to 0.01V, when a lithium is made into a counter electrode, and it can be more preferably inserted to 0.05V. Especially a desirable method is the method of inserting a lithium partially, in order to compensate the irreversible capacity which negative-electrode material has, and when a lithium is made into a counter electrode, it is the method of inserting to 0.3V. As an amount of insertion of a more concrete lithium, 0.005g - 0.03g - 0.2g 0.5g per 1g of negative-electrode material is 0.06g-0.15g especially preferably more preferably. When negative-electrode material is a metallic oxide, in the equivalent per one mol of metallic oxides, it is 0.5-4.0Eq, is 1-3.5Eq still more preferably, and is 1.2-3.2Eq especially preferably. When preliminary insertion of the lithium fewer than 1.2Eq is carried out at negative-electrode material, cell capacity is low, and when preliminary insertion of many lithiums is carried out from 3.2Eq, there is cycle nature degradation, and it is not desirable respectively.

[0046] The amount of lithium insertion can be arbitrarily controlled by the amount of the lithium piled



up on a negative-electrode sheet. Although it is desirable to use a lithium metal as a metal which made the lithium the subject, the thing of 90 % of the weight or more of purity is desirable, and 98% of the weight or more of especially a thing is desirable. Although it is desirable to pile up all over a sheet as a superposition pattern of the lithium on a negative-electrode sheet, since the lithium by which preliminary insertion was carried out at negative-electrode material is gradually diffused in negative-electrode material according to aging, the shape of not the whole sheet surface but a stripe and a frame and one of its disc-like partial superposition is also desirable. the superposition said here -- a negative electrode -- it means sticking by pressure the metallic foil which made the direct lithium the subject on the sheet which has a mixture and an auxiliary layer

[0047] Although 10 - 100% of the coverage of the metallic foil superposition in a negative-electrode sheet is desirable, 15 - 100% is more desirable, and especially 20 - 100% is desirable. 20% or less of case does not become uneven and have desirable preliminary insertion of a lithium. Furthermore, as for the thickness of the metallic foil which made the lithium the subject from a homogeneous viewpoint, it is desirable that it is 5-150 micrometers, its 5-100 micrometers are still more desirable, and especially its 10-75 micrometers are desirable. Handling atmosphere, such as cutting of the metallic foil which made the lithium the subject, and attachment, has the desirable bottom of -30 degrees-C or less the dried air - 80 degrees C or more or argon gas atmosphere of dew-points. In the case of a dried air, -40 degrees C or less -80 degrees C or more are still more desirable. Moreover, at the time of handling, you may use carbon dioxide gas together. Especially in the case of argon gas atmosphere, it is desirable to use carbon dioxide gas together.

[0048] If the electric conduction agent used for the mixture of the nonaqueous electrolyte rechargeable battery of this invention is an electronic-conduction nature material which does not cause a chemical change in the constituted cell, it is good anything. As an example, natural graphites, such as a flaky graphite, a scale-like graphite, and an earthy graphite, petroleum coke, Elevated-temperature baking objects, such as coal corks, celluloses, a saccharide, and a mesophase pitch, Graphite, such as artificial graphites, such as a vapor-growth graphite, acetylene black, Furnace black, KETCHIEN black, channel black, lamp black, Carbon black, such as thermal black, an asphalt pitch, a coal tar, Conductive metallic oxides, such as conductive whiskers, such as metal powders, such as conductive fiber, such as carbon materials, such as activated carbon, a meso fuze pitch, and the poly acene, and a metal fiber, copper, nickel, aluminum, and silver, a zinc oxide, and a potassium titanate, and titanium oxide, etc. can be mentioned. It is desirable when an aspect ratio uses five or more plate-like things in a graphite. In these, graphite and carbon black are desirable, the size of a particle has 0.01 micrometers or more and desirable 20 micrometers or less, and its particle (0.02 micrometers or more and 10 micrometers or less) is more desirable. These may be used independently and may use two or more sorts together. It is desirable when using together, and a 1-15-micrometer graphite particle is used together with carbon black, such as acetylene black. As for the addition to the binder layer of an electric conduction agent, it is desirable that it is 1 - 50 % of the weight to negative-electrode material or positive-electrode material, and it is especially desirable that it is 2 - 30 % of the weight. In carbon black or graphite, it is desirable that it is especially 3 - 20 % of the weight.

[0049] the nonaqueous electrolyte rechargeable battery of this invention -- an electrode -- a binder is used in order to hold a mixture As an example of a binder, the polymer which has polysaccharide, thermoplastics, and rubber elasticity is mentioned. As a desirable binder, starch, a carboxymethyl cellulose, A cellulose, a diacetyl cellulose, a methyl cellulose, a hydroxyethyl cellulose, Hydroxypropylcellulose, alginic-acid Na, a polyacrylic acid, Polyacrylic-acid Na, a polyvinyl phenol, a polyvinyl methyl ether, Polyvinyl alcohol, a polyvinyl pyrrolidone, a polyacrylamide, Water-soluble polymer, such as PORIHIDOROKISHI (meta) acrylate and a styrene-maleic-acid copolymer, Polyvinyl chloride, a polytetrafluoroethylene, a polyvinylidene fluoride, A tetrafluoroethylene-hexafluoropropylene copolymer, a vinylidene fluoride-tetrafluoroethylene-hexafluoropropylene copolymer, Polyethylene, polypropylene, an ethylene-propylene-diene terpolymer (EPDM), Sulfonation EPDM, a polyvinyl-acetal resin, a methylmetaacrylate, The acrylic-ester (meta) copolymer containing acrylic esters (meta), such as 2-ethylhexyl acrylate The polyvinyl ester copolymer containing vinyl

esters, such as an acrylic-ester-acrylonitrile copolymer and vinyl acetate, (Meta) A styrene-butadiene copolymer, an acrylonitrile-butadiene copolymer, A polybutadiene, neoprene rubber, a fluororubber, a polyethylene oxide, An emulsion (latex) or suspensions, such as a polyester polyurethane resin, a polyether polyurethane resin, a polycarbonate polyurethane resin, polyester resin, phenol resin, and an epoxy resin, can be mentioned. The latex of a polyacrylic ester system, a carboxymethyl cellulose, a polytetrafluoroethylene, and a polyvinylidene fluoride are mentioned especially. It is desirable to use what distributed minute powder in water, as for these binders, it is more desirable to use that whose average size of the particle in dispersion liquid is 0.01-5 micrometers, and it is desirable especially to use what is 0.05-1 micrometer.

[0050] these binders are independent -- or it can mix and use if there are few additions of a binder -- an electrode -- the holding power and cohesive force of a mixture are weak If many [ too ], electrode volume will increase and an electrode unit volume or the capacity per unit weight will decrease. The addition of a binder has 1 - 30 desirable % of the weight at such a reason, and 2 - 10 % of the weight is especially desirable.

[0051] In the constituted cell, a bulking agent can be used anything, if it is the fibrous material which does not cause a chemical change. Usually, fiber, such as olefin system polymer, such as polypropylene and polyethylene, glass, and carbon, is used. Although especially the addition of a filler is not limited, 0 - 30 % of the weight is desirable. The object known as an inorganic and organic solid electrolyte can be used for an ion electric conduction agent, and it is indicated by the term of the electrolytic solution for details. A pressure reinforcement agent is a compound which raises the internal pressure of a cell, and carbonates, such as a lithium carbonate, are the examples of representation.

[0052] The positive electrodes of the charge collector which can be used with the nonaqueous electrolyte rechargeable battery of this invention are aluminum, stainless steel, nickel, titanium, or these alloys, and negative electrodes are copper, stainless steel, nickel, titanium, or these alloys. The gestalt of a charge collector is a foil, an expanded metal, a punching metal, or a wire gauze. Especially, in a positive electrode, copper foil is desirable to an aluminum foil and a negative electrode.

[0053] Next, the composition of the positive/negative electrode in the nonaqueous electrolyte rechargeable battery of this invention is explained. a positive/negative electrode -- both sides of a charge collector -- an electrode -- it is desirable that it is the gestalt which applied the mixture In this case, even if the number of layers per one side is one layer, it may consist of more than two-layer. When the number of the layers per one side is two or more, a positive-active-material (or negative-electrode material) content layer may be more than two-layer. More desirable composition is the case where it consists of a layer containing a positive active material (or negative-electrode material), and a layer which does not contain a positive active material (or negative-electrode material). There are an interlayer between the protective layer for protecting the layer containing a positive active material (or negative-electrode material) and the divided positive-active-material (or negative-electrode material) content layer, an under coat between a positive-active-material (or negative-electrode material) content layer and a charge collector, etc. in the layer which does not contain a positive active material (or negative-electrode material), and these are named generically in this invention and it is called an auxiliary layer.

[0054] As for a protective layer, it is desirable that it is in either both positive/negative electrodes or a positive/negative electrode. In a negative electrode, when inserting a lithium in negative-electrode material within a cell, as for a negative electrode, it is desirable that it is the gestalt which has a protective layer. A protective layer consists of at least one layer, and may be constituted by a homotypic or two or more layers of a different kind. Moreover, you may be the gestalt which has a protective layer only on one side of the binder layers of both sides of a charge collector. These protective layers consist of particles, binders, etc. of water-insoluble nature. the electrode of the above-mentioned [ a binder ] -- the binder used in case a mixture is formed can be used As a particle of water-insoluble nature, a various conductive particle and the organic and inorganic particle which does not have conductivity substantially can be used. The solubility to the water of a water-insoluble nature particle has 100 ppm or less and an insoluble desirable desirable thing. The rate of the particle contained in a protective layer has

2.5 % of the weight or more and 96 desirable % of the weight or less, is more desirable, and is desirable. [ of 10 % of the weight or more and especially 93 % of the weight or less ] [ of 5 % of the weight or more and 95 % of the weight or less ]

[0055] As a conductive particle of water-insoluble nature, carbon particles, such as a metal, a metallic oxide, a metal fiber, a carbon fiber, carbon black, and a graphite, can be mentioned. In these water-insoluble conductive particles, a low thing has desirable reactivity with alkali metal, especially a lithium, and a metal powder and a carbon particle are more desirable. As electrical resistivity in 20 degrees C of the element which constitutes a particle, it is  $5 \times 10^9$ . Below  $\omega\text{-m}$  is desirable.

[0056] As a metal powder, the metal with which reactivity with a lithium cannot make a low metal, i.e., a lithium alloy, easily is desirable, and, specifically, copper, nickel, iron, chromium, molybdenum, titanium, a tungsten, and a tantalum are desirable. A needle, a column, a tabular, and massive any are sufficient as the form of these metal powders, 0.02 micrometers or more and 20 micrometers or less have a desirable overall diameter, and it is more desirable. [ of 0.1 micrometers or more and 10 micrometers or less ] That [ its ] to which the front face has not oxidized too much is desirable, and when having oxidized, as for these metal powders, heat-treating by reducing atmosphere is desirable.

[0057] The well-known carbon material used as an electrical conducting material used together as a carbon particle when an electrode active material is not conductivity conventionally can be used. concrete -- an electrode -- the electric conduction agent used in case a mixture is made is used

[0058] As a water-insoluble nature particle which does not have conductivity substantially, the impalpable powder of Teflon, SiC, alumimium nitride, an alumina, a zirconia, a magnesia, a mullite, a forsterite, and a steatite can be mentioned. When these particles may be used together with a conductive particle and used by 0.01 or more times of a conductive particle, and 10 or less times, they are desirable.

[0059] A positive (negative) electrode sheet can be created by applying, drying and compressing the mixture of a positive (negative) pole on a charge collector. A positive active material (or negative-electrode material) and an electric conduction agent are mixed, a binder (the suspension of resin fine particles or emulsion-like thing) and a dispersion medium are added, kneading mixture is carried out, succeedingly, it can distribute by stirring mixers, such as a mixer, a homogenizer, a dissolver, a planetary mixer, a paint shaker, and a sand mill, and the disperser, and manufacture of a mixture can be performed. Water is desirable although water or an organic solvent is used as a dispersion medium. In addition, you may add additives, such as a bulking agent, an ion electric conduction agent, and a pressure reinforcement agent, suitably. As for pH of dispersion liquid, in a negative electrode, 7-12 are desirable in 5-10, and a positive electrode.

[0060] Although an application can be performed by various methods, the reverse rolling method, the direct rolling method, the blade method, the knife method, the extrusion method, slide agglutination test, the curtain method, the gravure method, the bar method, the dipping method, and the squeeze method can be mentioned, for example. The method using an extrusion die and especially the method using a slide coating machine are desirable. As for an application, it is desirable to carry out the speed for 0.1-100m/. under the present circumstances, a mixture -- according to the liquid nature of a paste, and a drying property, the surface state of a good application layer can be obtained by selecting the above-mentioned method of application. When electrode layers are two or more layers, those things [ applying two or more layers simultaneously ] are desirable from viewpoints, such as manufacture of a uniform electrode, and a manufacturing cost. The thickness, length, and width of the application layer are decided with the size of a cell. The thickness of a typical application layer is 10-1000 micrometers in the state where it was compressed after dryness. The electrode sheet after an application is dried and dehydrated by hot blast, a vacuum, infrared radiation, far infrared rays, an electron ray, and the operation of the damp style. these methods are independent -- or it can combine and use. The range of drying temperature of 80-350 degrees C is desirable, and its range which is 100-260 degrees C is especially desirable. The moisture content after dryness has desirable 200 ppm or less, and its 100 ppm or less are more desirable. Although compression of an electrode sheet can use the press method generally adopted, a die-press method and its calender pressing method are especially desirable. Especially press \*\* is 10 kg/cm<sup>2</sup> - 3 t/cm<sup>2</sup>, although not limited. It is desirable. The press speed of the

calender pressing method has the amount of desirable 0.1-50m/. Press temperature has desirable room temperature -200 degree C.

[0061] The separator which can be used with the nonaqueous electrolyte rechargeable battery of this invention has large ion transmittance, and it has a predetermined mechanical strength, and that what is necessary is just an insulating thin film, as the quality of the material, olefin system polymer, fluorine system polymer, cellulose system polymer, a polyimide, nylon, a glass fiber, and an alumina fiber are used, and a nonwoven fabric, textile fabrics, and a microporosity film are used as a gestalt. Especially, as the quality of the material, the mixture of polypropylene, polyethylene, polypropylene, the mixture of polyethylene and polypropylene, and Teflon and the mixture of polyethylene and Teflon are desirable, and what is a microporosity film as a gestalt is desirable. Especially, the microporosity film whose thickness an aperture is 0.01-1 micrometer and is 5-50 micrometers is desirable. even if these microporosity films are independent films -- fine -- you may be the complex film which consists of more than two-layer [ from which properties, such as a configuration, density, etc. of a hole, and the quality of the material, differ ] For example, the complex film which made the polyethylene film and the polypropylene film rival can be mentioned.

[0062] The cell cans and cell lids which can be used with the nonaqueous electrolyte rechargeable battery of this invention are the steel board which performed nickel plating as the quality of the material, a stainless steel plate (SUS304, SUS304 L, SUS304 N, SUS316, SUS316 L, SUS430, SUS444 grade), the stainless steel plate (same as the above) which performed nickel plating, aluminum or its alloy, nickel, titanium, and copper, and are perfect circle form tubed one, ellipse form tubed one, square tubed, and rectangle tubed as a configuration. Especially when a sheathing can serves as a negative-electrode terminal, a stainless steel plate and the steel board which performed nickel plating are desirable, and when a sheathing can serves as a positive-electrode terminal, a stainless steel plate, aluminum, or its alloy is desirable. Any, such as a button, coin, a sheet, a cylinder, and an angle, are sufficient as the configuration of a cell can. A relief valve can be used for an obturation board as a cure of internal pressure elevation of a cell can. In addition, the method of putting in slitting can also be used for members, such as a cell can and a gasket. In addition, it may be equipped with the various safe elements (a fuse, bimetal, a PTC element, etc. as [ For example, ] an overcurrent-protection element) known from the former.

[0063] Metals (for example, iron, nickel, titanium, chromium, molybdenum, copper, aluminum, etc.) with electrical conductivity and those alloys can be used for the lead board used with the nonaqueous electrolyte rechargeable battery of this invention. A well-known method (electric welding of an example, a direct current, or an alternating current, laser welding, ultrasonic welding) can be used for the welding process of a cell lid, a cell can, an electrode sheet, and a lead board. The compound and mixture which are known from the former, such as asphalt, can be used for the sealing compound for obturation.

[0064] As the quality of the material, they are olefin system polymer, fluorine system polymer, cellulose system polymer, a polyimide, and a polyamide, from organic-solvent-proof nature and low-water-flow part permeability, the gasket which can be used with the nonaqueous electrolyte rechargeable battery of this invention has desirable olefin system polymer, and especially its polymer of a propylene subject is desirable. Furthermore, it is desirable that it is the block copolymerization polymer of a propylene and ethylene.

[0065] As for the cell assembled as mentioned above, it is desirable to perform aging processing. There are pretreatment, activation, after treatment, etc. in aging processing, and the cell which was excellent in a high charge-and-discharge capacity and cycle nature by this can be manufactured. Pretreatment is processing for equalizing a distribution of the lithium in an electrode, for example, the temperature control for making dissolution control of a lithium and a distribution of a lithium uniform, rocking and/or rotation processing, and arbitrary combination of charge and discharge are performed. Activation is processing for making a lithium insert to the negative electrode of the main part of a cell, and it is desirable to insert 50 - 120% of the amount of lithium insertion at the time of real use charge of a cell. After treatment is processing for fully carrying out activation, has this [ for a judgment / charge-and-discharge processing ], and can be arbitrarily combined with the preservation processing for making a

cell reaction uniform.

[0066] The cell of this invention is covered with a sheathing material if needed. As a sheathing material, there are heat-shrinkable tubing, an adhesive tape, a metal film, paper, cloth, a paint, a plastics case, etc. Moreover, the portion of sheathing discolored with heat in part at least is prepared, and you may make it the heat history in use known.

[0067] The cell of this invention constructs two or more in series and/or in parallel if needed, and is contained by the cell pack. You may prepare a safety circuit (circuit with the function which intercepts current if it acts as the monitor of the voltage of each cell and/or the whole group cell, temperature, the current, etc. and is required) besides safe elements, such as a right temperature coefficient resistor, a thermal fuse, a fuse, and/or a current interception element, in a cell pack. Moreover, the positive electrode of each cell and a negative-electrode terminal, the whole group cell and the temperature element child of each cell, the current element child of the whole group cell, etc. can also be prepared in a cell pack as an external terminal in addition to the positive electrode of the whole group cell, and a negative-electrode terminal. Moreover, you may build voltage conversion circuits (DC-DC converter etc.) in a cell pack. Moreover, it may fix by welding a lead board, and you may fix connection of each cell so that it can detach and attach easily with a socket etc. Furthermore, you may prepare display functions, such as cell remaining capacity, existence of charge, and a usage count, in a cell pack.

[0068] The cell of this invention is used for various devices. It is desirable to be especially used for a video movie, a carrying type videocassette recorder with a built-in monitor, a movie camera with a built-in monitor, a digital camera, a compact camera, a single-lens reflex camera, a disposable camera, a notebook sized personal computer, a note type word processor, an electronic notebook, a cellular phone, a cordless telephone, a mustached camber, a power tool, an electric mixer, an automobile, etc.

[0069]

[Example] Although an example is raised to below and this invention is explained in more detail, unless the main point of invention is exceeded, this invention is not limited to an example.

[0070] The cell of No.6 of the example 3 of a JP,7-312219, A public presentation official report was used for the cell for example-1 test. This cell is created as follows. Manufacture of a mixture was performed as follows. As a negative-electrode material,  $\text{SnSiO}_3$  ( $\text{SnO}$  and  $\text{SiO}_2$  are mixed, it calcinates for 1000 degree-C 12 hours, and synthetic jet mill grinds) 80 % of the weight, A scale-like graphite and acetylene black are mixed by the ratio of 9:5 as an electric conduction agent. 15% of the weight, the total electric conduction dose added 4 % of the weight and 1 % of the weight of carboxymethyl celluloses for the water distribution object of a styrene butadiene rubber as a binder further, kneaded water as a medium, and produced the slurry. This slurry was applied to both sides of copper foil with a thickness of 18 micrometers by the extrusion method, and carried out compression molding with the calender press machine after dryness, it cut to predetermined width of face and length, and the band-like negative-electrode sheet was produced. Positive-electrode material added  $\text{LiCoO}_2$  (commercial elegance is used) as a positive active material, and added 3 % of the weight of polytetrafluoroethylene water distribution objects, and 1 % of the weight of sodium polyacrylate as a binder further 87 % of the weight, 6 % of the weight of scale-like graphites, and 3 % of the weight of acetylene black, water was pressed [ applied the slurry which kneaded as a medium and was obtained by the method same to both sides of an aluminum foil with a thickness of 20 micrometers as the above, dried it, and ] and cut, and the band-like positive-electrode sheet was produced The above-mentioned negative-electrode sheet and a positive-electrode sheet are each the dew-point after carrying out spot welding of the lead board of nickel and aluminum to an edge, respectively. - Dehydration dryness was carried out in dry air 40 degrees C or less for 210-degree-C 2 hours. Furthermore, the laminating was carried out in the order of a dried [ dehydration ] positive-electrode sheet (moisture content of 15 ppm), a fine porosity polypropylene film separator (Celgard 2400), a dried [ dehydration ] negative-electrode sheet (moisture content of 20 ppm), and a separator, this was involved in, and it wound in the shape of a swirl by the opportunity.

[0071] It contained with the iron closed-end cylindrical cell can which serves this winding object as a negative-electrode terminal and which performed nickel plating. Furthermore, it is 1 mol/l as an electrolyte.  $\text{LiPF}_6$  (2:8 capacity mixed liquor of ethylene carbonate and diethyl carbonate, 8 ppm of

hydrogen fluoride contents) was poured into the cell can. the cell lid which has a positive-electrode terminal after applying to the cell can side of the gasket made from polypropylene (annealing -- unsettled) the sealing compound which makes asphalt a subject -- this gasket -- minding -- the cylindrical cell (A-1) was produced in total In addition, the positive-electrode terminal connected the positive-electrode sheet and the cell can with the negative-electrode sheet by the lead terminal beforehand. The cell (A-2) was created completely like the cell (A-1) except using the gasket made from polypropylene which annealed at 105 degrees C beforehand for 1 hour. It created the cell ten [ at a time ], respectively. Cell can insertion of an electrode, above-mentioned electrolytic-solution pouring, and above-mentioned cell can obturation were performed on the conditions not more than dew-point minus 50 degree C, and the cell was manufactured.

[0072] They are these cells 1 mA/cm<sup>2</sup> The charge and discharge test (4.1V-2.7V) of 250 cycles was performed with current density. The averages of each cell of the 300th service capacity were 310 mAh/g and 315 mAh/g. the place which disassembled these cells of three each and carried out the fixed quantity of the amount of isolation hydrogen fluoride -- a cell A-1 and A-2 -- it was both 50-60 ppm

[0073] After charging the remainder of the above-mentioned cell further and discharging to 3.2V, it saved for three years in the room of about 25-degree-C60% humidity. when these cells of five each were disassembled and the fixed quantity of the amount of isolation hydrogen fluoride was carried out, the cell (A-2) was 150-260 ppm, and the cell (A-1) was 330-780 ppm When the charge and discharge test of the remainder of a cell (A-1) was carried out, charge and discharge were possible for the cell (A-2) to the ability to have not carried out charge and discharge. As mentioned above, the effect of the cell (A-2) of this invention using the gasket which annealed is clear. Moreover, the fixed quantity value of the amount of isolation hydrogen fluoride is as low as 90-110 ppm, and the cell (A-4) using the gasket which was smaller than 100-150 ppm and the cell (A-2) as for the amount of isolation hydrogen fluoride of the cell (A-3) using what applied the sealing compound also inside the gasket, and gave two coats of a sealing compound again after sealing-compound application dryness further had the smallest width of face of measured value.

[0074] The relation between the amount of hydrogen fluoride detected from the example -2 electrolytic solution and a cell performance is explained.

[positive electrode -- a mixture -- creation] of a paste

Positive active material; LiCoO<sub>2</sub> (what put into the alumina crucible what was mixed with a lithium carbonate and 4 oxidization 3 cobalt by the mole ratio of 3:2, calcinated at 900 degrees C for 8 hours, and was further cracked at them at the speed of 2 degrees C/m at the temperature of temperature up *Perilla frutescens* (L.) Britton var. *crispa* (Thunb.) Decne. after carrying out the temperature up to 750 degrees C and carrying out temporary quenching to them at 2 degrees C/m for 4 hours, among air and.) The electric conductivity of the dispersion liquid when distributing 50g of centriole child size 5micrometer powder in 100ml water 0.6 mS/m, A specific surface area according [ pH ] to 10.1 and a nitrogen adsorption process 0.42m<sup>2</sup> / g 200g and acetylene black 10g It mixes and continues with a homogenizer. as a binder 2-ethylhexyl acrylate, an acrylic acid, and the water distribution object (50 % of the weight of solid-content concentration) of the copolymer of acrylonitrile 8g, carboxymethyl-cellulose solution of 2 % of the weight of concentration -- 60g -- adding -- kneading mixture -- carrying out -- further -- water -- 50g -- adding -- a homogenizer -- stirring mixture -- carrying out -- a positive electrode -- a mixture -- the paste was created

[0075] [negative electrode -- a mixture -- creation] of a paste

Negative-electrode active material; SnSi<sub>0.5</sub> B<sub>0.3</sub> P<sub>0.2</sub> Cs<sub>0.05</sub>aluminum 0.1O<sub>3</sub>.13 (11.0g of tin protoxides) 4.2g of pyrophosphoric-acid tin, 1.1g of boron oxides, 0.83g of cesium carbonates, Blend dryly 3.1g of silicon dioxides, put into the crucible made from an alumina, and a temperature up is carried out to 1000 degrees C by part for bottom 15-degree-C/of argon atmosphere. After calcinating at 1100 degrees C for 12 hours, what lowered even to the room temperature by part for 10-degree-C/, and was taken out from the firing furnace is collected. the thing and 4.5 micrometers of mean particle diameters ground with the jet mill It is the object which has the broadcloth peak which has the peak near 28 degree with 2theta value in the X-ray diffraction method using CuK alpha rays, and the crystalline



diffraction line was not looked at by 40 degrees or more 70 degrees or less with 2theta value. It mixes with 30g and a homogenizer. 200g and an electric conduction agent (artificial graphite) -- the thing and water which furthermore added 50g of carboxymethyl-cellulose solution of 2 % of the weight of concentration, and 10g of polyvinylidene fluorides as a binder, and were mixed -- 30g -- adding -- further -- kneading mixture -- carrying out -- a negative electrode -- a mixture -- the paste was created [0076] [Creation of negative-electrode protective-layer paste] alumina 85g and 9g of artificial graphites were added to 300g of carboxymethyl-cellulose solution of 2 % of the weight of concentration, kneading mixture was carried out, and it created.

[0077] the positive electrode created by [creation of positive-electrode and negative-electrode electrode sheet] above -- a mixture -- after applying at both sides of an aluminum foil charge collector with a thickness of 20 micrometers by coverage 280 g/m<sup>2</sup> of the positive-active-material conversion per one side and drying by the blade coating machine, compression molding of the paste was carried out so that the thickness of the sheet after compression might be set to 280 micrometers with a roller-press machine. Then, it judged in the predetermined size and the band-like positive-electrode sheet was created. Furthermore, it heated at the far-infrared heater in the dry box (dew-point; dry air -50 degrees C or less), dehydration dryness was enough carried out at the electrode temperature of about 250 degrees C, and the positive-electrode sheet (moisture content of 13 ppm) was created. the same -- both sides of a 18-micrometer copper foil charge collector -- a negative electrode -- a mixture -- the paste and the negative-electrode protective-layer paste were applied this time -- a negative electrode -- a mixture -- the paste applied to the charge collector side so that a negative-electrode protective-layer paste might become the best layer. For the coverage of the negative-electrode material conversion per one side, the coverage of 90 g/m<sup>2</sup> and a protective layer is 15 g/m<sup>2</sup>. The thickness of the sheet after compression with a roller-press machine created the negative-electrode sheet (moisture content of 20 ppm) which is 90 micrometers. The lithium metal (99.8% of purity) of the shape of a strip of paper of 6mm width of face is stuck on both sides of this negative-electrode sheet in a 10mm pitch, and the amount of lithiums is 10 g/m<sup>2</sup>. It was made to become. a mixture -- the coverage of the upper lithium was 60%

[0078] In [electrolytic-solution manufacture] argon atmosphere, 65.3g diethyl carbonate was put into the polypropylene container of a 200 cc thin mouth, and the 22.2g ethylene carbonate was dissolved small quantity every, taking care that solution temperature does not exceed 30 degrees C to this. Next, 0.4g LiBF<sub>4</sub> and 12.1g LiPF<sub>6</sub> It dissolved in the above-mentioned polypropylene container small quantity every in order, respectively, taking care that solution temperature does not exceed 30 degrees C. The obtained electrolytic solution was a transparent and colorless liquid by specific gravity 1.135. As for a part for 5 ppm (Kyoto electron make tradename MKC-210 type car RUF shear water measurement equipment measurement) and a free acid, the moisture content obtained the 8 ppm (the bromthymol blue is used as an indicator, a neutralization titration is carried out using decinormal NaOH solution, and it measures) criteria electrolytic solution in adjustment, removing moisture as much as possible. Various kinds of electrolytic-solution a-h from which the nitrogen gas which has humidity is blown into this criteria electrolytic solution, and a moisture content differs was made.

[0079] The cylinder cell as shown in [creation of cylinder cell] drawing 1 was produced. The laminating was carried out to the order of the separator made from a positive-electrode sheet (3) microporosity polyethylene film (the Ube Industries make, EF4500) (4), a negative-electrode sheet (2), and a separator (4), and this was wound in the shape of a whorl. After containing with the iron closed-end cylindrical cell can (1) which performed nickel plating which serves this winding object as a negative-electrode terminal, the above-mentioned electrolytic solution was poured in into the cell can. the cell lid (13) which has a positive-electrode terminal after applying to the gasket made from polypropylene (7) the sealing compound which makes asphalt a subject -- this gasket (7) -- minding -- the cylindrical cell was produced in total. In addition, the positive-electrode terminal connected the positive-electrode sheet and the cell can with the negative-electrode sheet by the lead terminal beforehand. the inside of drawing -- (5) -- a lower electric insulating plate and (6) -- an up electric insulating plate and (8) -- a positive-electrode lead and (9) -- in a PTC ring and (13), a cell lid (it serves as a positive-electrode terminal) and (15) show a welding plate, and (16) shows [ an explosion-proof valve element and (10) / a current

isolating switch (the 10a 1st flow object, the 10b 2nd flow object,, 10c insulating ring  
 [0080] The above-mentioned cell was performed at the room temperature, preliminary charge was performed after 12-hour neglect and under the fixed current of 0.1A for 1 hour, and, subsequently aging was carried out for ten days by the 50-degree C basis. Next, because of activation, it is 2 mA/cm<sup>2</sup>. It charged under the room temperature to 4.2V. Furthermore, aging between three days of maintenance was given to 55 degrees C for the cell in the state of charge. It is this cell Charge final-voltage 4.2V (open-circuit voltage), discharge-final-voltage 2.8V, and 2 mA/cm<sup>2</sup> Charge and discharge were carried out with current density, and initial service capacity was measured. Furthermore, the cycle was repeated, service capacity was measured after the 100 cycle end, and it asked for the maintenance factor to initial service capacity. Moreover, the cell after measurement was disassembled and the fixed quantity of the amount of isolation hydrogen fluoride was carried out. The result was shown in the following table.  
 Table -1 Experimental-result electrolytic solution Battery discharge capacity maintenance factor The amount a of isolation hydrogen fluoride 95% 24ppmb(s) 95 48c 95 90d 94 140e 92 190f 90 280g 85 360h 79 The cell by which the amount of isolation hydrogen fluoride detected from the electrolytic solution within a cell exceeds 300 ppm from the result beyond 500 has a remarkably low service-capacity maintenance factor, and it turns out that it is required to control [ 200 ppm or less ] to 150 ppm or less especially preferably [ it is desirable and ].

[0081]

[Effect of the Invention] The nonaqueous electrolyte rechargeable battery of this invention is high capacity, is excellent in shelf life and has the advantage that a life is long.

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[Translation done.]

**\* NOTICES \***

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**CLAIMS**

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[Claim(s)]

[Claim 1] The nonaqueous electrolyte rechargeable battery characterized by being the amount in which the amount of the free acid contained in this nonaqueous electrolyte does not exceed 300 ppm after pouring in the nonaqueous electrolyte containing lithium salt in the nonaqueous electrolyte rechargeable battery of the closed mold which comes to obturate after passing through three years after obturation.

[Claim 2] The nonaqueous electrolyte rechargeable battery according to claim 1 characterized by being lithium salt in which the lithium salt contained in nonaqueous electrolyte contains a fluorine.

[Claim 3] The nonaqueous electrolyte rechargeable battery according to claim 1 or 2 characterized by the free acid contained in this nonaqueous electrolyte being hydrogen fluoride.

[Claim 4] The nonaqueous electrolyte rechargeable battery according to claim 1, 2, or 3 with which the gasket used for the obturation section is characterized by carrying out a forming postheat treatment.

[Claim 5] A nonaqueous electrolyte rechargeable battery given in any 1 term of the claims 1-4 characterized by the amount of the free acid which nonaqueous electrolyte before pouring into a cell can contains being 100 ppm or less.

[Claim 6] A nonaqueous electrolyte rechargeable battery given in any 1 term of the claims 1-5 characterized by the moisture content which the electrode group before inserting in a cell can contains being 300 ppm or less.

[Claim 7] A nonaqueous electrolyte rechargeable battery given in any 1 term of the claims 1-6 characterized by for the environment at the time of cell can insertion of this electrode group, electrolytic-solution pouring, and cell can obturation having been managed by less than [ dew-point minus 50 degree C ], and assembling it.

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[Translation done.]